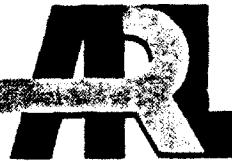


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Effect of Powder Processing on Slip Casting Parameters and Sintering of Si₃N₄-Y₂O₃-SiO₂

Gary Gilde and George Gazza

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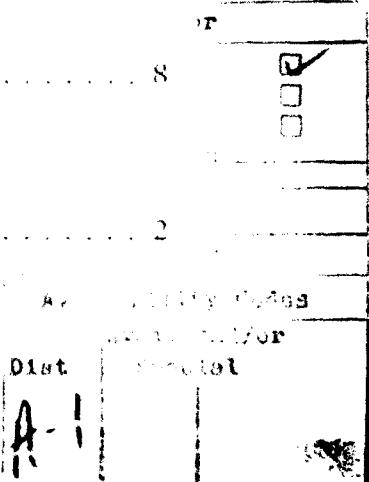
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Introduction

Slip casting of Si_3N_4 - Y_2O_3 - SiO_2 compositions is difficult because at a pH less than 8, Y_2O_3 forms positive counter ions of 3+ by the dissolution of its hydroxide [1] and at a pH greater than 9 the solubility of silica increases rapidly [2]. Trivalent yttria ions can flocculate the slip at low concentrations. In order to keep the concentration of yttria ions as low as possible, it is necessary to keep the pH above 8. When the silica becomes soluble it is difficult to produce a homogeneous distribution of the silica and the viscosity of the slip also increases. Maintaining the pH between 8 and 9 is difficult. When Si_3N_4 is milled or aged in water, the pH usually rises above 9 due to the dissociation of Si_3N_4 to form ammonia. Typical strategies for dispersing silicon nitride often include raising the pH above 9 to take advantage of its high negative surface charge at high pH. With slip casting compositions with high silica content, this is not advantageous.

The goal of this study was to determine if Si_3N_4 - Y_2O_3 - SiO_2 compositions can be formulated and slip cast by oxidizing the surface of the silicon nitride to uniformly distribute the SiO_2 and increase the negative zeta potential of the Si_3N_4 , while using commercial polyelectrolyte dispersants to adjust the pH of the suspension to between 8.3 and 8.8. It was believed that oxidizing the silicon nitride surface would aid in dispersing it and would yield a high quality "green" body with improved distribution of silica on the surface of the powder, higher "green" densities, and a more uniform pore distribution.

Experimental

Two silicon nitride powders were used in this study: UBE-E-10 from UBE Industries (Japan), and LC-12-SX from Hermann Starck (Germany). The silicon nitride was oxidized at 1000°C in 1/2-inch-high alumina crucibles for the selected time. The oxygen content, after oxidation, was determined by inert gas fusion. For the standard composition, a three-hour oxidation of the UBE-E-10 was selected to adjust the silica content and the yttria was varied from 5.5 w/o to 6.5 w/o. The yttria/silica composition was controlled to provide a molar ratio of approximately 1:2.

Five anionic polyelectrolytes (see Table 1) and an organic amine (3-amino-1-propanol) were tested as dispersants. Viscosity versus dispersant level curves (demand curves) were then established for the five polyelectrolytes. A 1 w/o addition of 3-amino-1-propanol was used, as suggested by Starck. Slips were milled for 24 hours with various dispersant levels and the viscosity was measured using a shear rate of 15.84s^{-1} . Based on initial demand curves, Nopcosperse 644A was selected for further investigation. Viscosity versus dispersant level curves for 60 w/o, 65 w/o, and 70 w/o slips were developed using UBE and Starck silicon nitride powders in both oxidized and the as-received condition. Either yttria and silica or yttria and alumina were used as sintering aids. Silicon nitride, alumina, or nylon covered steel milling media were used with either distilled or distilled deionized water.

Table 1. Material specifications of dispersants (manufacturer specifications)

Name	Distributor	% Active ingredient	Molecular weight	Active ingredient	pH
Darvan C	R. T. Vanderbilt, Inc.	25	10 to 16000	Ammonia polymethacrylate	7.5 to 9.0
Darvan 821A	R. T. Vanderbilt, Inc.	40	≈6000	Ammonia polyacrylate	7.0
Gradol 250A	Grader Chemical Co.	25	Unknown	Ammonia polymethacrylate	1% solution 9.5 to 10.5
Alcosperse 244	ALCO Chemical	40	3200	Ammonia polyacrylate	7.0 to 9.0
Nopcosperse 644A	Henkel Corporation	35	Unknown	Proprietary blend ammonium polyelectrolyte	8.0 to 8.5

Results And Discussion

Oxidation of Starting Powder

Figure 1 shows the oxygen content of the two silicon nitride powders after oxidation at 1000°C for various times. The Starck powder oxidizes much more rapidly. This would be expected because of its greater surface area. Both powders exhibit parabolic oxidation behavior shown when an exponential plot of the weight percent of oxygen gained is graphed versus oxidation time in Figure 1. When the plots are normalized for surface area, the slopes of each plot do not change significantly. This suggests that the kinetics of oxidation are somewhat different for each powder.

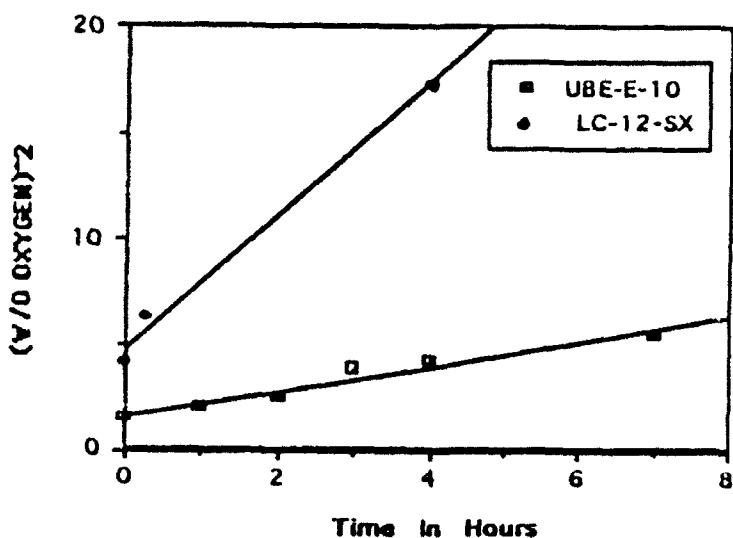


Figure 1. Weight percent oxygen versus oxidation time at 1000°C.

Another factor that may be influencing the oxidation rates is the ratio of surface to bulk oxygen. The LC-12-SX, which is made by the direct nitridation of silicon, has an oxygen content of 2.0% approximately 45% of which is surface oxygen distributed over a surface area of $20 \text{ m}^2/\text{g}$. UBE-E-10 has a total oxygen content of 1.3% and a surface area of $13 \text{ m}^2/\text{g}$. UBE does not specify the amount of surface oxygen versus the total oxygen. It is suspected that the oxygen is principally distributed on the surface. A thicker layer of surface silica, a greater amount of substitutional oxygen, and a lower surface area could explain the different oxidation rates.

Effect of Oxidation of Starting Powders on Sintering

During the sintering experiments, the as-received LC-12-SX would not densify with 6.0 w/o yttria whereas the UBE-E-10, oxidized for three hours, with 6.0 w/o yttria, was easily sintered to full density. The total oxygen content for the as-received LC-12-SX and the UBE-E-10 with a three-hour oxidation was 2.0%. When the LC-12-SX was oxidized for 15 minutes and 6 w/o yttria was added, it could be sintered to 92% of theoretical density. It appears that there is a critical amount of surface silica needed to activate the sintering for this composition. When UBE-E-10 (as-received plus 6 w/o yttria) was fired it would not sinter to full density. When it was oxidized for three hours it was easily sintered to full density with yttria contents ranging from 5.5 w/o to 6.5 w/o. Compacts that were pressure cast using oxidized UBE with 6 w/o yttria could be sintered to full density at 1850°C for 180 minutes with a nitrogen overpressure of 4 MPa using a two-step sintering cycle. This is a low firing temperature for a refractory composition fired with a 4 MPa overpressure of nitrogen.

By oxidizing the silicon nitride to provide the silica for this composition it is expected that the silica will be more homogeneously distributed than if it is added as a powder. Since the silica is uniformly distributed on the surface of the silicon nitride powder, it should promote a uniform distribution of the liquid phase to enhance sintering at lower temperatures. In comparing the sintering behavior of the slip case compacts made with oxidized silicon nitride to the sintering behavior of compacts formed from cold pressing and CIPing as-received silicon nitride with silica additions from previous studies [3], it appeared the oxidized silicon nitride sintered at lower temperatures. It is recognized that the slip cast bodies also had higher densities and were probably less agglomerated.

Selection of Dispersant

After generating the oxidation curves for the powders, a standard slip casting procedure was developed. The first step was to select a dispersant. Five anionic polyelectrolytes and 3-amino-1-propanol were tested. A 1.0 w/o addition of 3-amino-1-propanol, as suggested by Starck, produced a good slip and appeared to be promising; however, sintered pieces made from compacts using this dispersant had a layered appearance. The lower portion of the compact was white, the top portion was gray. The pH of slips made with the 3-amino-1-propanol was 10.3. At this high pH, it is believed that the silica became soluble and was concentrated on the bottom portion of the compact as the piece was dewatered in the pressure caster. This area

became the white area on firing. For this reason, it was decided to use a anionic polyelectrolyte and work at pH levels between 8.3 and 8.9.

Figure 2 compares the demand curves for the five dispersants tested using 50 w/o slips of LC-12-SX with 5.0 w/o yttria and 5.0 w/o alumina. The slips were milled in 500 ml Nalgene bottles with 250 grams of alumina milling media. Table 1 shows the characteristics of each dispersant. As can be seen in Figure 2, the two polyacrylates behave similarly giving identical minimum viscosities at the same w/o dispersant. The two polymethacrylates were more effective dispersants for the silicon nitrides used in this study, producing significantly lower viscosities than the polyacrylates. Again, both polymethacrylates appeared to behave similarly. The Nopcosperse, although not producing a lower viscosity than either of the polymethacrylates tested, was more resistant to over-deflocculation. For this reason, the Nopcosperse was selected over the other dispersants.

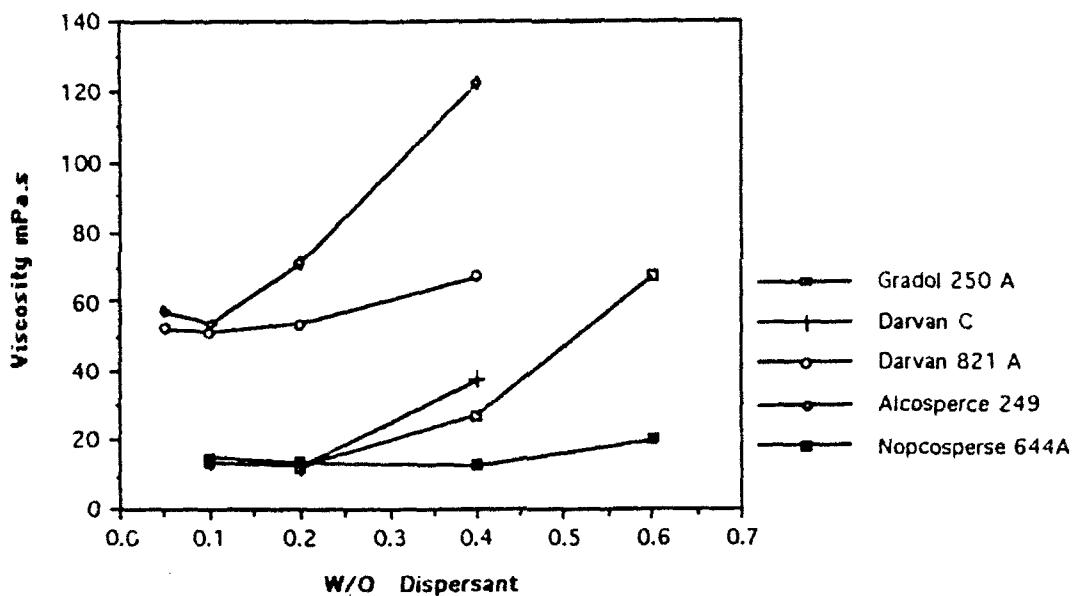


Figure 2. Demand curves for the five dispersants tested with 50 w/o slips of LC-12-SX with yttria and alumina sintering aids dispersed in distilled water and milled with alumina milling media.

Effect of Powder Oxidation on the Properties of the Slip

A comparison of Figures 2 and 3 shows that as the solids loading of the slip is increased from 50 w/o to 65 w/o, the slips become more sensitive to over-deflocculation. Figure 3 shows that oxidizing the UBE-E-10 did not result in a lower viscosity slip than the as-received powder. This is in conflict with the results found by Greil [4]. However, in his study, Greil used Stark's LC-12 silicon nitride. When LC-12-SX powder was oxidized in this study, it also resulted in a lower viscosity slip. Starck's LC-12 and LC-12-SX are made by the direct nitridation of silicon while

the UBE powder is synthesized by a wet chemical process. Differences in behavior may be related to the different powder synthesis methods. Greil [5] proposed that there is a minimum silica layer thickness that is needed to shield the silicon nitride surface. It is plausible that if most of the oxygen is on the surface, the surface layer of silica may exceed this minimum thickness even for a silicon nitride with low oxygen content. This does not explain the difference in the pH of slips produced with oxidized silicon nitride and as-received silicon nitride. Slips made from oxidized silicon nitride typically had a pH of 8.3, while the as-received silicon nitride resulted in slips with a pH above 9. If the as-received silicon nitride surface is shielded with a silica layer, it would be expected that the pH of the slip should be similar to that of the slip produced with oxidized silicon nitride, unless adsorbed ammonia is responsible for the rise in pH. UBE-E-10 silicon nitride was heated to 1000°C in nitrogen to remove any surface impurities. The behavior of the UBE-E-10 held at 1000°C for three hours was the same as the as-received silicon nitride; therefore, the difference in pH is not believed to be the result of adsorbed surface impurities.

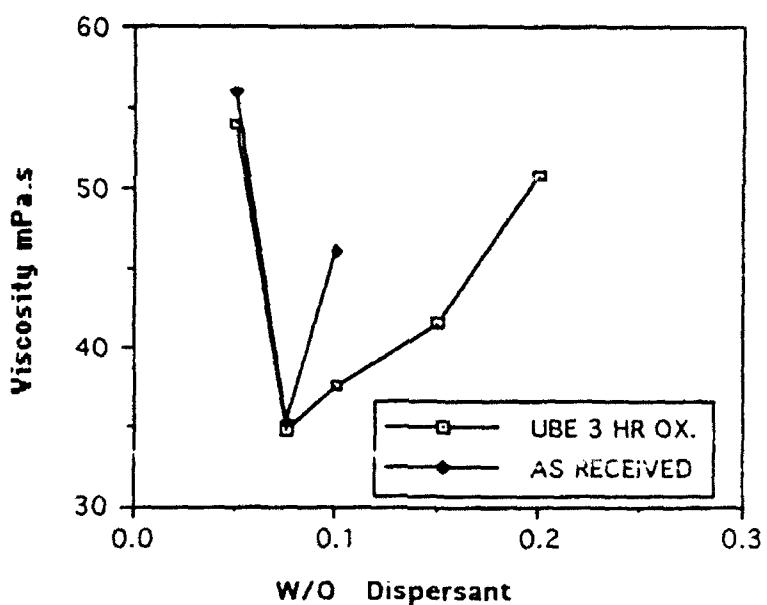


Figure 3. Viscosity versus dispersant level for two 65 w/o slips prepared with either as-received UBE-E-10 plus silica additions or oxidized UBE-E-10; dispersed in distilled deionized water and milled with nylon media.

It can be seen in Figure 4 that oxidizing the LC-12-SX significantly changes its behavior. The oxidized slip at 60 w/o is much more resistant to over-deflocculation, with the minimum viscosity at 0.1 w/o dispersant. The as-received LC-12-SX is more sensitive to over-deflocculation, with a minimum viscosity at 0.3 w/o dispersant. The minimum viscosity for the as-received powder slip is also significantly higher than that of the oxidized LC-12-SX.

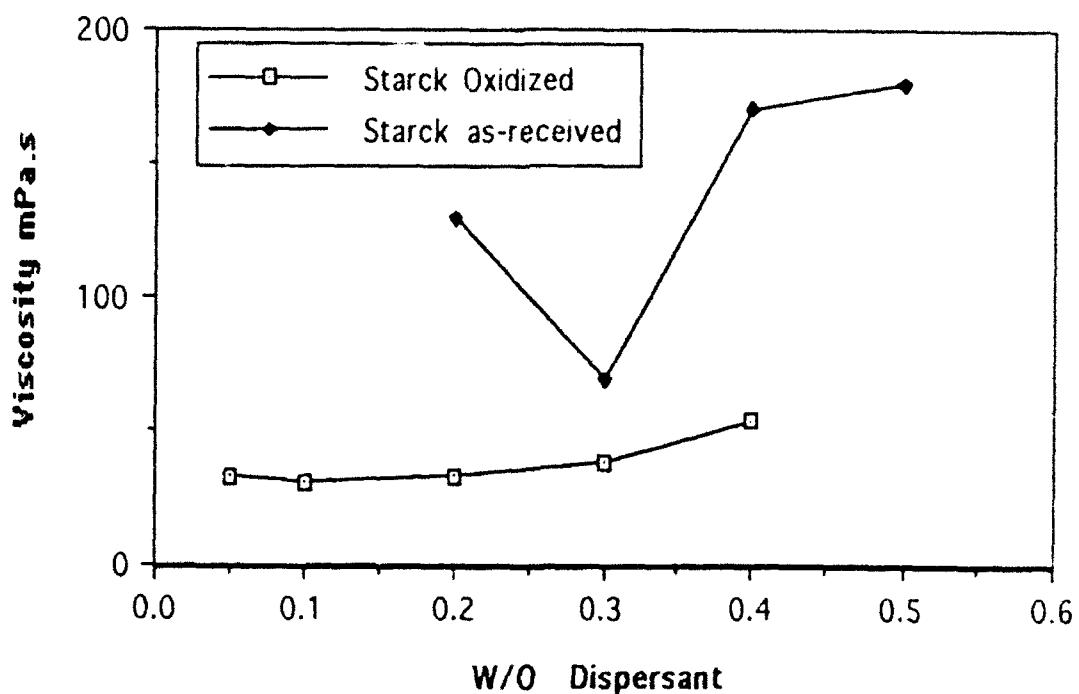


Figure 4. Slip viscosity versus w/o dispersant for 60 w/o slips with either as-received Starck LC-12-SX or Starck LC-12-SX oxidized for 15 minutes with 5 w/o yttria and 5 w/o alumina as sintering aids. The slips were prepared with distilled, deionized water using alumina milling media.

Oxidized UBE-E-10 acts as a strong acid lowering the initial pH of a slurry of silicon nitride and distilled deionized water to 4.5. When the nylon media was used for milling, it took 50 hours for the pH to rise to 7. The dramatic lowering of the pH when oxidized silicon nitride is used is believed to be associated with the high silica surface. It is hypothesized that at 1000°C, where the silicon nitride is oxidized, a dehydroxylated pyrogenic silica surface is produced. This would explain why the oxidized silicon nitride acts as an acid [6]. The nature of this dehydroxylated pyrogenic silica surface and its behavior in aqueous suspensions merits further investigation. It is known that the charge density of a dehydroxylated pyrogenic silica are higher than for a rehydrated surface. Below a pH of 9 or 10 a dehydroxylated surface acts opposite to that of a hydroxylated surface [6].

For a slip of the standard composition, the pH is 8.3 after 24 hours of milling. The pH of the water and dispersant alone is also 8.3. When the oxidized silicon nitride is put in water containing the dispersant, the pH is lowered. The pH is 7 after 72 hours of mixing on a shaker with no milling media. When the 6 w/o yttria is added, the pH rises to 8.4 in minutes. This mix was then milled for 24 hours using nylon media. The pH after 24 hours remained at 8.4. Both the oxidized silicon nitride and the yttria have a major effect on the pH of the slip. When slips with 70 w/o solids or higher are prepared, it was found that it was necessary to add at least some of the yttria before adding the silicon nitride or it was not possible to get an initial solids loading of 65 w/o.

Influence of Milling Media

During the course of this investigation, it became apparent that the use of different milling media were affecting the viscosity. Originally, the nylon covered steel media were used because it was reasoned that they would eliminate chipping of the milling media. It was also observed that the nylon-coated steel milling media produced slips with lower viscosities. This can be seen by comparing the 65 w/o slips in Figure 5. Demand curves for 65 w/o slips prepared with UBE-E-10 oxidized for three hours with 6 w/o yttria using distilled deionized water were milled with either alumina or nylon covered steel milling media. The nylon milled slips gave consistently lower viscosity. The pH of the slips milled with the nylon media ranged between 8.3 and 8.5, whereas the pH of the slips milled with alumina media ranged from 8.7 to 8.9. It is believed that the nylon covered steel media does not remove the surface layer of silica and the slip pH remains stable. The alumina media fractures the silicon nitride particles or abrades the surface exposing the Si_3N_4 to water where it reacts to form ammonia. The hydrolysis reactions are discussed by Greil [1]. The increase in viscosity could be due to the increase in pH, the increase of ions in the slip, or the increase in surface area due to milling.

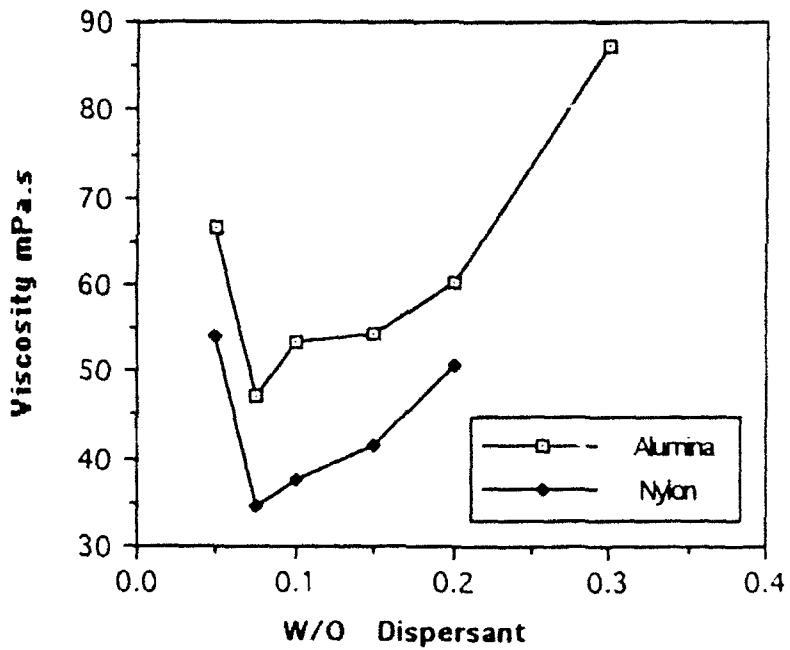


Figure 5. Viscosity versus dispersant level for 65 w/o slips with UBE-E-10 oxidized for three hours dispersed in distilled deionized water milled with nylon or alumina milling media.

Figure 6 shows that the pH versus milling time for UBE silicon nitride powder as-received and oxidized (for three hours), is dependent on the milling media. The slips were milled with either silicon nitride milling media or nylon covered media. Additionally, pH versus milling time is also shown for as-received Starek silicon nitride milled with nylon media. The graph illustrates that for slips milled with the nylon covered media, the pH does not rise above 9, even after 200 hours of milling.

When the silicon nitride milling media is used, it causes the pH to rise above 10 in under 150 hours. The milling media plays an important role in determining the final pH of the slip.

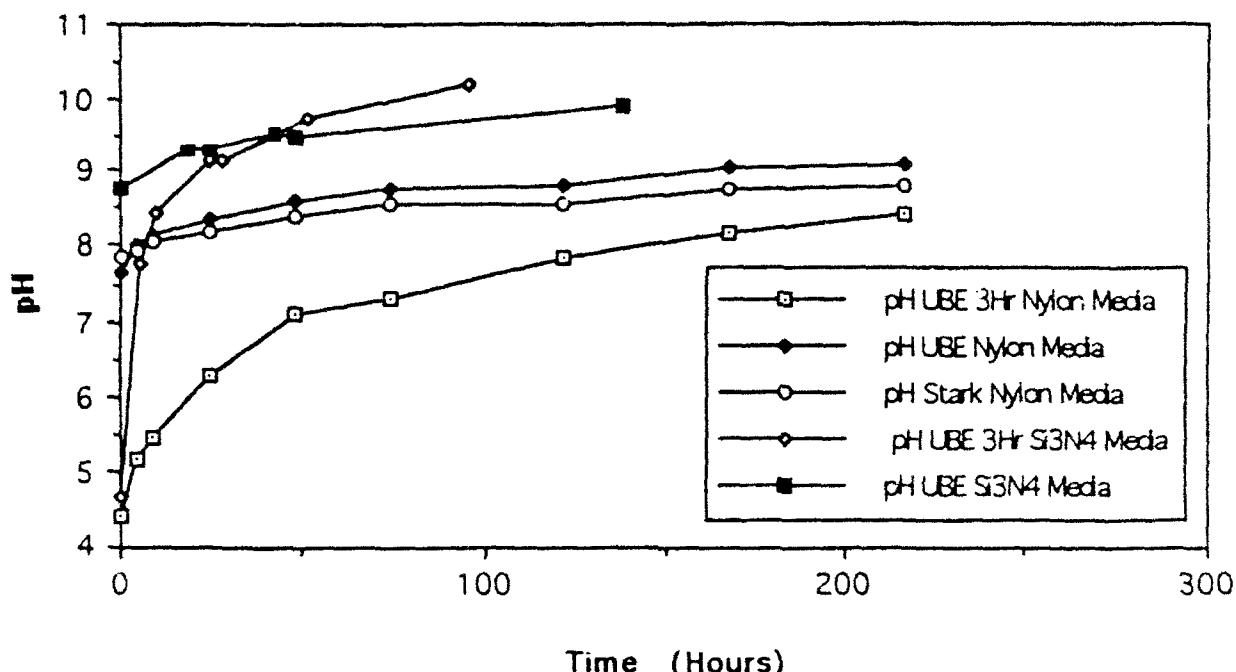


Figure 6. Milling time versus pH for as-received UBE-E-10, LC-12-SX, or UBE-E-10 oxidized for three hours milled with nylon and silicon nitride milling media

Unfortunately, after extended use, the polymer coating on the steel ball begins to wear off of the substrate and the slurry begins to pick up iron. Future work is planned to find a way of coating the milling media that is more wear resistant.

Conclusions

1. Under the slip conditions studies, the Nopcosperse 644A polyelectrolyte was found to be the most effective dispersant for the compositions investigated.
2. Oxidizing both the UBE and Stark silicon nitride starting powders enhanced the sinterability of the compositions studied.
3. Reductions in slip viscosity were observed when oxidized Stark LC-12-SX powder was used instead of as-received powder. This effect was not observed with oxidized UBE powder.
4. The use of nylon-coated milling media produced lower slip viscosities than ceramic milling media when used to prepare slips with the oxidized silicon nitride.

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